

## PATENT SPECIFICATION

Application Date : Feb. 24, 1930. No. 6082 / 30.

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PROVISIONAL SPECIFICATION.



### Improvements in and relating to Coating and Impregnating Compositions.

We, IMPERIAL CHEMICAL INDUSTRIES LIMITED, of Imperial Chemical House, Millbank, London, S.W. 1, a Company incorporated under the laws of Great Britain, and WILLIAM BAIRD, of Crumpsall Vale Chemical Works, Blackley, Manchester, a British Subject, do hereby declare the nature of this invention to be as follows:—

In our co-pending application No. 39,596/29 we have shown that polyhydric-alcohol-polybasic-acid resinous composition, commonly known and hereinafter referred to as glyptal resins (the word "glyptal" being a Registered Trade Mark) can be used in the manufacture of emulsions which have advantages over solutions in organic solvents with respect to cost, freedom from fire hazard etc., when utilised as coating and impregnating compositions.

We have found and our invention is based on the discovery, that we may incorporate rapid thermo hardening resins such as phenol formaldehyde resoles and urea formaldehyde condensation products in the emulsions and we thus obtain compositions which harden much more rapidly when heated in bulk or e.g. in the form of a film or coating, than similar compositions containing only glyptal resins. In our copending application 39,596/29 we have described the preparation of aqueous emulsions from modified glyptal resins in which the usual glyptal resin formation is effected in the presence of a resin of the urea formaldehyde type but in this case the modifying resin has entered, previous to emulsification, into what is believed to be chemical combination with the glyptal resin, so that the resulting product is not a mechanical mixture but rather a chemical union of the two. In the present instance on the other hand no such preliminary chemical combination of the glyptal resin and the modifying urea formaldehyde condensation product is effected but mixed emulsions of glyptal resin and urea formaldehyde product or of glyptal resin and phenol formaldehyde resole are employed. It is known that rapid hardening products can be obtained

by using mixtures of glyptal resins and thermo hardening resins but hitherto these have been used only in the form of solid mixtures or solutions in organic solvents. Trouble is frequently experienced in the latter case in finding suitable solvents or solvent mixtures to dissolve the resins or resin mixtures and give a satisfactory film on evaporation. These and the like difficulties can be avoided by the use of the emulsion obtained by the method of our invention.

The value of our invention may be appreciated by considering the use of urea formaldehyde condensation products with glyptal resins. Generally speaking the urea formaldehyde resins are soluble in water, glycol esters and chlorhydrins only. These latter two are very expensive and glyptals are insoluble in water. By our invention however the glyptal and urea formaldehyde resin may be incorporated together by emulsifying the glyptal in an aqueous solution of the urea formaldehyde resin using a suitable emulsifying agent.

We do not limit ourselves to any specific glyptal, emulsifying agent, natural or synthetic resin, to the inclusion of organic solvents in our compositions or to any particular method of carrying out our invention. Furthermore we may add other ingredients such as plasticisers, dyestuffs, catalysts, such as may be necessary. As examples of synthetic resins we may use the urea formaldehyde condensation products in their water soluble stage and phenol formaldehyde resole dispersed in water by means of ammonium oleate but we do not limit ourselves to these. Furthermore, we may incorporate the starting materials of the synthetic resin in the emulsion e.g. urea and paraformaldehyde and cause them to react before or after application of the composition.

Our invention is illustrated, but not limited by the following examples, in which the parts are by weight.

#### EXAMPLE I.

8 parts of urea formaldehyde resin made from 2 mols. formaldehyde and 1 mol. urea using boric acid as catalyst, and

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0.25 parts of ammonia are mixed with 95 parts of water. 40 parts of glyptal resin prepared as in Example I of our application No. 26,411/29 is dissolved in 32 parts solvent naphtha, and 28 parts of butyl alcohol, to which solution 4 parts oleic acid is added. The two solutions are mixed with vigorous stirring. An oil in water emulsion is formed which can be applied by any known means to e.g. rubber, leather cloth, or the like and hardened by heating for two hours at 115° C.

#### EXAMPLE II.

7 parts of dimethylol urea, 0.4 parts casein, 0.25 parts ammonia, and 100 parts water are dissolved up together. 40 parts glyptal resin as in Example I is dissolved in a mixture of 28 parts butyl alcohol and 32 parts solvent naphtha. The two solutions are emulsified together and applied as in Example I.

#### EXAMPLE III.

40 parts of glyptal resin as in Example

I are dissolved in a mixture of 32 parts solvent naphtha and 28 parts butyl alcohol and 4 parts oleic acid are added. 0.25 parts of ammonia and 0.4 parts of casein are dissolved in 100 parts of water. By vigorous stirring or other suitable means an emulsion is prepared from these two solutions. To the emulsion 68 parts of an aqueous dispersion of phenol formaldehyde resole containing 25% resole is added and thoroughly commingled by similar means.

The emulsion is of the oil in water type and may be applied by known means to rubber, rubber leather cloth and similar materials and hardened by heating 1 hour at 115° C.

Dated the 24th day of February, 1930.

E. C. G. CLARKE,  
Imperial Chemical House,  
Millbank, London, S.W.1,  
Solicitors for the Applicants.

### COMPLETE SPECIFICATION.

#### Improvements in and relating to Coating and Impregnating Compositions.

We, IMPERIAL CHEMICAL INDUSTRIES LIMITED, of Imperial Chemical House, Millbank, London, S.W. 1, a Company incorporated under the laws of Great Britain, and WILLIAM BAIRD, of Crumpsall Vale Chemical Works, Blackley, Manchester, a British Subject, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

In our co-pending application No. 39,596/29 we have shown that polyhydric-alcohol-polybasic-acid resinous composition, commonly known and hereinafter referred to as glyptal resins (the word "glyptal" being a Registered Trade Mark) can be used in the manufacture of aqueous emulsions which have advantages over solutions in organic solvents with respect to cost, freedom from fire hazard, etc., when utilised as coating and impregnating compositions.

It is well known that "glyptal" resins harden more slowly under the application of heat than resins of the urea- and phenol-formaldehyde types, the latter being well recognised as rapid-hardening resinous compositions. It has been proposed to improve the hardening properties of "glyptal" resins by mixing them with phenol formaldehyde resins

in solution in organic solvents or in the solid state. It has also been proposed to obtain products exhibiting improved thermo-hardening properties by combining "glyptal" resins with urea formaldehyde condensation products.

Trouble is frequently experienced in the former case in finding suitable solvents or solvent mixtures to dissolve the resins or resin mixtures and give a satisfactory film on evaporation. These and the like difficulties can be avoided by the use of the emulsions obtained by the method of our present invention.

According to the present invention we produce composite emulsions capable of giving coatings exhibiting improved thermo-hardening properties by bringing together an aqueous emulsion of a "glyptal" resin and a phenol-formaldehyde resole or a urea formaldehyde condensation product dissolved or dispersed in water. The emulsification of the "glyptal" resin may be performed prior to or simultaneously with the production of the composite emulsion.

In our co-pending application 39,596/29 referred to above we may have described the preparation of aqueous emulsion from products containing "glyptal" resins and urea-formaldehyde condensation products already in a state of chemical combination. In the present

instance on the other hand no such preliminary chemical combination of the glyptal resin and the urea formaldehyde condensation product is effected but composite emulsions of glyptal resin and urea formaldehyde product or of glyptal resin and phenol formaldehyde resole are produced.

The value of our invention may be appreciated by considering the use of urea formaldehyde condensation products with glyptal resins. Generally speaking the urea formaldehyde resins are soluble in water, glycol esters and chlorohydrins only. These latter two are very expensive and glyptals are insoluble in water. By one embodiment of our invention (as described in the illustrative example given below) however the glyptal and urea formaldehyde resin may be incorporated together by emulsifying the glyptal in an aqueous solution of the urea formaldehyde condensation product.

We do not limit ourselves to any specific "glyptal" resin or rapid thermo-hardening synthetic resin; nor do we limit ourselves to the use of any specific solvent, emulsifying agent, or protective colloid in the preparation of emulsions according to our invention. Furthermore, we may add plasticisers, colouring matters, fillers, etc., as is desirable.

The term "dissolved" here is intended to mean that a true solution is produced, and the term "dispersed" that a suspension or colloidal solution is produced.

Our invention is illustrated, but not limited, by the following examples, in which the parts are by weight.

#### EXAMPLE 1.

8 parts of urea formaldehyde resin made from 2 mols. formaldehyde and 1 mol. urea using boric acid as catalyst, and 0.25 parts of ammonia are mixed with 95 parts of water. 40 parts of glyptal resin prepared by heating 31 parts of glycerol, 74 parts of phthalic anhydride and 75 parts of castor oil for 3-4 hours at 190-200° C. with stirring are dissolved in 32 parts solvent naphtha, and 28 parts of butyl alcohol, to which solution 4 parts oleic acid are added. The two solutions are mixed with vigorous stirring. An emulsion of the oil in water type is formed which can be applied by any known means to e.g. rubber, leather cloth, or the like and hardened by heating for two hours at 115° C.

#### EXAMPLE 2.

7 parts of dimethylol urea, 0.4 parts casein, 0.25 parts ammonia, and 100 parts water are dissolved up together. 40 parts glyptal resin as in Example 1 are dissolved in a mixture of 28 parts butyl

alcohol and 32 parts solvent naphtha. The two solutions are emulsified together and applied as in Example 1.

#### EXAMPLE 3.

40 parts of glyptal resin as in Example 1 are dissolved in a mixture of 32 parts solvent naphtha and 28 parts butyl alcohol and 4 parts oleic acid are added. 0.25 parts of ammonia and 0.4 parts of casein are dissolved in 100 parts of water. By vigorous stirring or other suitable means an emulsion is prepared from these two solutions. To the emulsion 68 parts of an aqueous dispersion of phenol formaldehyde resole containing 25% resole is added and thoroughly commingled by similar means.

The emulsion is of the oil in water type and may be applied by known means to rubber, rubber leather cloth and similar materials and hardened by heating 1 hour at 115° C.

#### EXAMPLE 4.

6 parts of oleic acid are thoroughly mixed with 40 parts of glyptal resin containing 46% of linseed oil and 10 parts of ammonia (S.G. 0.88) dissolved in 70 parts of water at 70° C. added. The mixture is stirred vigorously until the resin has been emulsified. 40 parts of the urea-formaldehyde resin made by refluxing 85 parts dimethylolurea with 100 parts of 20% formaldehyde for 1 hour are added to the glyptal emulsion and mixed thoroughly therewith.

The product when applied to a glass plate and stoved for 1 hour at 120° gives a fairly hard film with a slightly frosted appearance.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. A composition of matter suitable for coating and impregnating which comprises a composite aqueous emulsion of "glyptal" resin and a rapid thermo-hardening resin or substances capable of giving such rapid thermo-hardening resin.

2. A composition of matter as claimed in claim 1 in which the rapid thermo hardening resins are phenol formaldehyde resoles and/or urea formaldehyde condensation products.

3. A process for the manufacture of coating and impregnating compositions which consists in emulsifying "glyptal" resins which may or may not be dissolved in organic solvents and incorporating aqueous solutions or dispersions of thermo-hardening resins with the said emulsions either during or after emulsification.

4. A process of manufacture as claimed in claim 3 in which the rapidly thermo hardening resin is a formaldehyde-urea condensation product and/or a phenol  
5 formaldehyde resole.

5. A process of manufacture of coating and impregnating compositions sub-

stantially as described with reference to the examples.

Dated the 24th day of November, 1930.

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